

SEP. 20. 2004 1:34PM

3M DENTAL PRODS 260 2B 09

NO. 2559 P. 2

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PATENT  
Docket No. 57160US002**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicant(s): Darren T. CASTRO et al. ) Group Art Unit: 1775  
Serial No.: 10/034,642 )  
Confirmation No.: 9543 ) Examiner: Gwendolyn Blackwell Rudasill  
Filed: December 28, 2001 )  
For: POLYCRYSTALLINE TRANSLUCENT ALUMINA-BASED CERAMIC  
MATERIAL, USES AND METHODS

**Declaration under 37 C.F.R. §1.131**

Assistant Commissioner for Patents  
Mail Stop AF  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

We, Darren T. Castro and Richard P. Rusin, declare and say as follows:

1. We are the inventors of the subject matter of the claims presently pending in the above-identified U.S. Patent Application Serial No. 10/034,642, filed December 28, 2001.
2. I, Darren T. Castro, am a Technical Manager at 3M, St. Paul, Minnesota.
3. I, Richard P. Rusin, am a Product Development Specialist at 3M, St. Paul, Minnesota.
4. We have reviewed the above-entitled U.S. Patent Application.

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P. 3

Declaration under 37 C.F.R. §1.131

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Serial No.: 10/034,642

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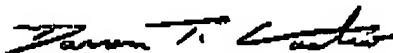
For: POLYCRYSTALLINE TRANSLUCENT ALUMINA-BASED CERAMIC MATERIAL, USES AND METHODS

5. Prior to May 19, 2000, one of us, Darren T. Castro, reduced to practice a polycrystalline translucent aluminum oxide ceramic material having an average grain size of no greater than 1.0 micron, and a method of making the same. This invention is evidenced, for example, by the redacted notebook pages marked Exhibits A-C.


6. Specifically, Exhibit A describes a method for preparing a sample of polycrystalline aluminum oxide ceramic material (TM-DAR 1250) with low grain growth ("g g") by hot isostatic pressing ("HIPing") a sintered article at 1250°C. Exhibit B describes the sample as "translucent / clear." The scanning electron micrograph of sample TM-DAR 1250 illustrated in Exhibit C shows an average grain size of no greater than 1.0 micron.

6. We hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the likes so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

20 Sep 04  
Date

  
Darren T. Castro

9/20/04  
Date

  
Richard P. Rusin

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3M DENTAL PRODS 260 2B 09T REPRODUCE

NO. 2559 P. 4

NOTEBOOK NO.

PROJECT NO. -Redacted-

SUBJECT: -Redacted-

DATE: -Redacted-

-Redacted-

Want to prepare a batch of cast TM-DAR to try HIPing at 1250 C or lower to push to full density with less  
2 g. than got at 1300 C for 2 hrs. In looking at the last 2 batches of transvaped TM-DAR, first batch  
ground 137% (1861 g total cut vs 1363 g for C321) of C321 while 2<sup>nd</sup> did only 126% (1673 g total cut vs  
1322 g for C321). Differences in prep were use of foamkill in first batch, higher burnout T (690 C in 1<sup>st</sup> vs.  
630 C in 2<sup>nd</sup>), and the amount of water used. Follow first batch procedures directly as shown below:

Bottle weighs 101.03 g. Added 170.62 g of TM-DAR, 0.24 g of NHC, 63.74 g of water, and 1 drop of  
foamkill. Was very agglomerated/pasty. Added another 73.44 g of water without any beneficial effect.  
Added another 0.29 g of NHC. Dispersed it all very nicely. Sonicated for 2 hrs and then poured out into  
pan. Did transvap. Again this appeared to be too much water and not quite enough powder. Produced 169  
g of flakes. Burn out in Linberg in air at 690 C overnight.

-Redacted-

AUTHOR'S FULL NAME or INITIALS -Redacted-

DATE: -Redacted-

WITNESS'S FULL NAME or INITIALS -Redacted-

DATE: -Redacted-

(READ AND UNDERSTOOD)

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3M DENTAL PRODS 260 2B 09JT REPRODUCE

NO. 2559, P. 5GE

Exhibit B

NOTEBOOK NO.

- Redacted -

1 PROJECT NO.

- Redacted -

SUBJECT:

- Redacted -

DATE:

- Redacted -

Object

5 Refer

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- Redacted - TM-DAR nh some 2 hrs, transvap # 3, 1230 C, 1.5 hrs 20 C/min r and c in  
air, HIP at 1250 C for 30 min at 30 ksi

Deg C= H2O Dens  
72 22.222222 0.9977

| WD     | WSS    | WS     | p         | % p    |
|--------|--------|--------|-----------|--------|
| 0.3155 | 0.2356 | 0.3155 | 3.9396039 | 99.48% |
| 0.253  | 0.1885 | 0.253  | 3.9134589 | 98.82% |
| 0.1681 | 0.1258 | 0.1684 | 3.9369336 | 99.42% |
|        |        |        | 3.9299988 | 99.24% |

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30

No real difference in ore and post HIP density, but these flakes are translucent / clear.

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TMDAR HIP 1250 C - very nice densification. Smallest grains seen yet with powder Al2O3 materials!

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AUTHOR'S FULL NAME or INITIALS

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DATE:

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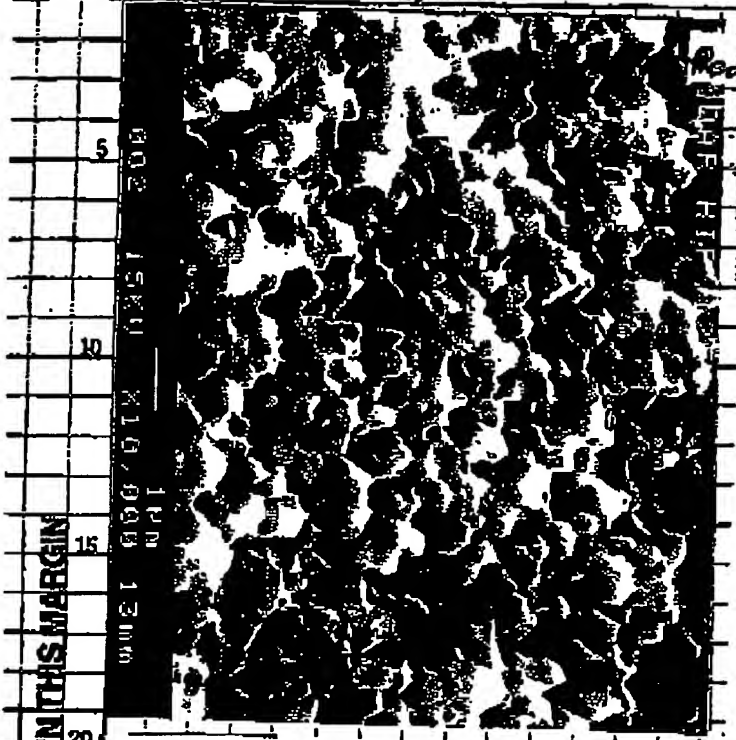
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1. PROJECT NO.

- Redacted -

SUBJECT:

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AUTHOR'S FULL NAME or INITIALS \_\_\_\_\_

- Redacted -

DATE: - Redacted -

WITNESS'S FULL NAME or INITIALS \_\_\_\_\_

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